

<p><b>90-248023/33</b>      A41 E17      <b>BADI 04.02.89</b>  <b>BASF AG</b>      *EP-382-050-A  04.02.89-DE-903363 (16.08.90) C07c-29/13 C07c-31/20  <b>Butane-1,4-diol(s) and pentane-1,5-diol(s) prodn. by</b>  <b>hydrogenation of corresp. lactone(s) in presence of a catalyst contg.</b>  <b>cobalt and manganese etc..</b>  <b>C90-107070</b>    R18E CH DE FR GB IT LI NL)</p>	<p>Prepn. of alkanols of formula (I) is by hydrogenation of lactones of formula (II) at 100-350°C and 20-350 bar in the presence of a catalyst (III) contg. Co and at least one of the elements Mn, Cu and P;</p> <div style="text-align: center;"> <math display="block">  \begin{array}{c}  \text{R}^1 \quad \text{R}^3 \\    \quad   \\  \text{HO} - \text{C} - \text{C} - \text{X} - \text{CH}_2\text{OH} \quad (I) \\    \quad   \\  \text{R}^2 \quad \text{R}^4  \end{array}  </math>   <math display="block">  \begin{array}{c}  \text{R}^3 \quad \text{R}^4 \\    \quad   \\  \text{C} - \text{C} - \text{X} \\    \quad   \quad   \\  \text{O} \quad \text{O} \quad \text{O}  \end{array}  </math>   (II) </div> <p>X = -CR<sup>5</sup>R<sup>6</sup>- or -CR<sup>5</sup>R<sup>6</sup>-CR<sup>7</sup>R<sup>8</sup>-; and  R<sup>1</sup>-R<sup>6</sup> = H, OH, or 1-4C alkyl, alkoxy or hydroxyalkyl (R<sup>1</sup>, R<sup>3</sup>, R<sup>5</sup> and R<sup>7</sup> can also = 5-7C cycloalkyl).</p>
<p>A(1-E14) E(10-E4B, 10-E4C) N(2-D, 3-D, 3-E, 4-B)</p>	<p><b>ADVANTAGE</b>  Use of (III) enables the prodn. of (I) in good yield even at temps. in the lower part of the above temp. and pressure ranges; prior-art catalyst present problems w.r.t. toxic effluent disposal (high-Cr catalysts), radioactive waste (Th-contg. catalysts) or low selectivity.</p> <p><b>MORE SPECIFICALLY</b>  (III) contains Co and at least two of the elements Mn, Cu, P and Mo or (III) contains Co and at least three of the elements Mn, Cu, P, Mo and Na; at least 40 wt. % of the active catalyst material consists of Co, pref. with up to 10 wt. % Mn, up to 10 wt. % P and up to 1 wt. % Na as other active components, esp. pref. 40-80 wt. % Co, 3-7 wt. % Mn, 0.1-3 wt. % P and 0.01-0.5 wt. % Na, or with up to 10 wt. % Mn, up to 30 wt. % Cu, up to 5 wt. % Mo, up to 10 wt. % P and up to 1 wt. % Na, esp. pref. 40-60 wt. % Co, 3-7 wt. % Mn, 0.1-3 wt. % P, 12-20 wt. % Cu, 0.5-5 wt. % Mo and 0.01-0.5 wt. % Na.</p> <p><b>DETAIL</b>  (II) is, e.g. γ-butyrolactone, δ-valerolactone or alkyl-substd. derivs. thereof, etc.; (II) is produced by impregnating a support (silica, alumina, TiO<sub>2</sub>, active EP-382050-A+</p>

carbon, zeolite, etc.) with aq. solns. of the corresp. metal salts and P cpds., drying, and calcining at 400-600°C to form metal oxides and phosphoric acid; the oxides are reduced to the corresp. metals with hydrogen before use.

**EXAMPLE**

A catalyst contg. 52.5 wt. % Co, 5.1 wt. % Mn, 15.3 wt. % Cu, 2.2 wt. % Mo, 1.1 wt. % P and 0.1 wt. % Na was produced as above (extruded pellets, 2mmx3mm dia.) and reduced with hydrogen for 72 hrs. at 310°C and 250 bar; hydrogenation was carried out in a tube reactor at 208°C and 60 bar, using 1212 l/hr. hydrogen; under these conditions  $\gamma$ -butyrolactone was hydrogenated at 98g/hr. (1.14 mol.) to give 51.6g/hr. (0.6 mol.) unchanged starting material, 35.1 g/hr. (0.39 mol.) butane-1, 4-diol, 0.15 g/hr. (0.002 mol.) THF, 0.8g g/hr. (0.01 mol.) n-butanol, 0.4g/hr. (0.006 mol.) n-propanol and 0.4g/hr. (0.022 mol.) water. (7pp1712HWDwgNo0/0).  
(G) ISR: US3848003 US3478112 US4141930 DE2321101  
EP-304696

EP-382050-A

BEST AVAILABLE COPY